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Adhesion of resin composite to enamel and dentin: A methodological assessment

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Adhesion of resin composite to enamel and dentin: a methodological assessment

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ABSTRACT

This study compared the impact of four test methods on adhesion of resin composite to enamel and dentin. Human molars ($N = 54$) were randomly assigned to test the adhesion of resin composite material (Quadrant Universal LC) using one of the following test methods: (a) macroshear test (SBT; $n = 16$), (b) macrotensile test (TBT; $n = 16$), (c) microshear test (μ SBT; $n = 16$) and (d) microtensile test (μ TBT; $n = 6$). In a randomized manner, buccal or lingual surfaces of each tooth, were assigned as enamel or dentin substrates. Enamel and dentin surfaces were conditioned using an etch-and-rinse adhesive system (Syntac Classic). After storage (24 h, 37 °C), bond tests were conducted in a Universal Testing Machine (1 mm/min) and failure types were analyzed. Data were analyzed using Univariate and Tukey's, Bonferroni tests ($\alpha = 0.05$). Two-parameter Weibull modulus, scale (m) and shape (0) were calculated. Test method ($p < 0.001$) and substrate type ($p < 0.001$) significantly affected the results. When testing adhesion of resin composite to enamel, SBT (25.9 ± 5.7)^a, TBT (17.3 ± 5.1)^{a,c} and μ SBT (27.2 ± 6.6)^{a,d} test methods showed significantly higher mean bond values compared to μ TBT (10.1 ± 4.4)^b ($p < 0.05$). Adhesion of resin composite to dentin did not show significant difference depending on the test method (12 ± 5.7 – 20.4 ± 4.8 ; $p > 0.05$). Only with SBT, significant difference was observed for bond values between enamel (25.9 ± 5.7) and dentin (12 ± 5.7 ; $p < 0.05$). Weibull distribution presented the highest shape values for enamel- μ SBT (29.7) and dentin- μ SBT (22.2) among substrate-test combinations. Regardless of the test method, cohesive failures in substrate were more frequent in enamel (19.1%) than in dentin (9.8%).

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Introduction

Advances in adhesive technologies during the last few decades introduced large number of resin-based materials for direct and indirect dental application that could be adhered to enamel or dentin. Reliable adhesion of the resin composites to enamel becomes particularly important in bonding brackets to non-prepared enamel surfaces in orthodontics or bonding surface-retained restorations or fixed dental prosthesis (FDP) where no macromechanical

retention is available. Likewise, durable adhesion to dentin is required for minimal invasive applications after tooth preparation as a consequence of caries removal, for restoring tissue loss due to trauma and bonding partial to full coverage crowns or FDPs.

Adhesion to enamel is typically achieved after etching enamel with H_3PO_4 that creates a highly micro-retentive surface that is easily wetted by hydrophobic resin-based adhesives [1]. The adhesive resin then penetrates the etched surface through capillary action and subsequent polymerization of the resin facilitates micromechanical adhesion. Most commercially available enamel etching agents have a concentration ranging between 30 and 40%. When the concentration is less, the dicalcium phosphate dihydrate precipitate forms in the enamel surface that is very difficult to remove by rinsing [1]. For orthodontic applications, enamel tissue removal is not needed but for some applications in reconstructive dentistry, minimal room has to be created for the material that eventually necessitates the removal of surface enamel using mechanical methods such as the use of diamond burs, disks or airborne particle abrasion. The next step after micromechanical roughening of the enamel is the application of the adhesive resin where the conditioned surface provides the foundation for better wettability of the adhesive resin and the following resin composite [2,3].

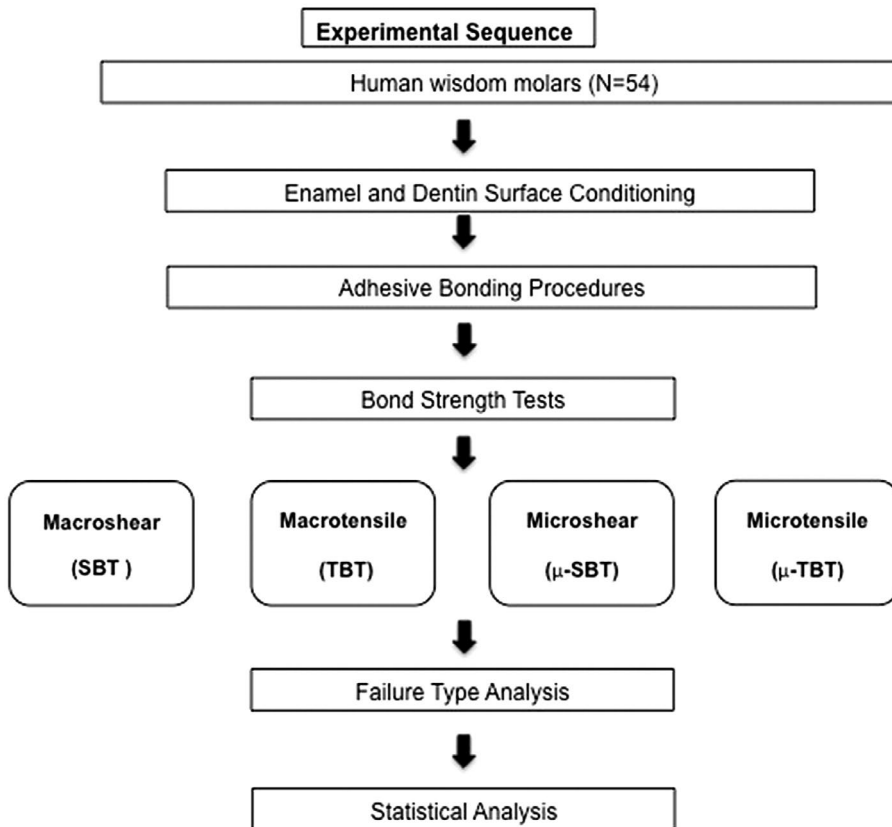
Adhesion to dentin on the other hand, is best achieved using 'etch-and-rinse' adhesive systems that rely on the application of adhesive monomers to acid-etched dentin [4–6]. The use of simplified self-etching, self-priming agents that contain hydrophilic and acidic monomers, acidic molecules, diluent monomers, photoinitiators, and solvents with usually low pH could also simultaneously etch the dentin and allow infiltration of the adhesive monomers into the dentin [7]. However, previous studies have shown that self-etch adhesives may result in lower bond strength to dentin and result in more permeability compared to etch-and-rinse adhesive systems [7]. Demineralization of the dentin substrate and penetration of the resin monomers create micromechanical retention that further contributes to the overall adhesion [4–6].

Meta-analysis in the field of adhesion in dentistry signified that depending on the test method employed and the variation in chemical compositions, bond strength of resin based materials to dentin between 9 and 45.3 MPa [8]. Today, an increased number of adhesive materials are being offered for clinical use. Neither ethically, nor technically it is possible to test their performance in randomized controlled clinical trials. Therefore, preclinical evaluations help to rank their adhesive properties. For this purpose several testing methodologies, (i.e. macroshear, microshear, macrotensile, and microtensile tests) have been suggested for evaluation of the bond strength of resin-based materials to dental tissues. Technically in macro bond tests, the bonded area is more than 3 mm² and in micro test set-ups it is less than 3 mm² [9]. According to the Griffith's theory [10], the tensile strength of the uniform materials decreases when the specimen size is increased. In that respect, the type of test method also affects the achieved bond strength and thereby, ranking of resin based materials. Unfortunately, to date, limited number of studies compared several test methods in one study or used enamel as a control substrate when testing dentin adhesives [8,11].

Since the adhesive joints in clinical applications are subjected to both shear and tensile form of forces during chewing, the objectives of this study were to evaluate the adhesion of resin composite to enamel and dentin using macro- and micro-shear and tensile adhesion methods and to evaluate the failure types after debonding. The null hypotheses tested were that bond strength results would not show significant difference depending on the test method and the substrate type.

Table 1. The brands, manufacturers and chemical compositions of the main materials used in this study.

Brand	Manufacturer	Chemical composition
Total etch	Ivoclar vivadent	37% phosphoric acid, water
Syntac primer	Ivoclar vivadent	Acetone 25–50%, Triethylenglycoldimethacrylate 10–<25%, Polyethylenglycoldimethacrylate 3–<10%, Maleic acid (3–<10%)
Syntac adhesive	Ivoclar vivadent	Polyethylenglycoldimethacrylate 25–50%, Glutaraldehyde 3–<10%
Heliobond	Ivoclar vivadent	bis-GMA (50–100), Triethylenglycoldimethacrylate (25–50%)
Qadrant universal LC	Cavex, Haarlem, The Netherlands	Feldspar 20–<25%, bis-phenol A Diglycidyl Methacrylate (bis-GMA) 10–20%, Silica, fused (0.1–<=2.5%)

**Figure 1.** Flow-chart showing experimental sequence and allocation of groups.

Materials and methods

The brands, types, manufacturers and chemical compositions of the materials used in this study are listed in Table 1. Distribution of experimental groups based on the substrate type and test methods and sequence of experimental procedures are presented in Figure 1.

Specimen preparation

Human wisdom molars ($N = 54$), were collected and kept in distilled water at 5 °C until the experiments. All teeth used in the present study were extracted for reasons unrelated to this project. Written informed consent for research purpose of the extracted teeth was obtained by all donors prior to extraction according to the directives set by the National Federal Council. Ethical guidelines were strictly followed and irreversible anonymization was performed in accordance with State and Federal Law [12–14]. After tissue remnants were removed with a scaler (H6/H7; Hu-Friedy, Chicago, IL), teeth were stored in 0.5% Chloramin T for 2 weeks. The roots of the teeth were embedded in a polyvinyl chloride (PVC) mould using auto-polymerizing acrylic resin (Scandiquick, Scandia, Hagen, Germany) allowing their buccal and lingual surfaces exposed for bonding purposes. Number of specimens for each tests were as follows: macroshear test (SBT; $n = 16$), macrotensile test (TBT; $n = 16$), microshear test (μ SBT; $n = 16$) and microtensile test (μ TBT; $n = 6$, $n_{\text{sticks-enamel}}: 52$, $n_{\text{sticks-dentin}}: 43$). In a randomized manner, buccal or lingual surfaces of each tooth, were assigned as enamel or dentin substrates. Enamel and dentin surfaces were prepared and conditioned according to the technical specification ISO/TS 11405 as follows [15]:

Enamel preparation

The enamel surfaces of each tooth were conditioned with etch and rinse adhesive system (Syntac Classic, Ivoclar Vivadent, Schaan, Liechtenstein) according to the manufacturer's recommendations. Firstly, the enamel was etched for 60 s with 37% H_3PO_4 , rinsed for s and then gently air-dried for 5 s. Then, adhesive resin (Heliobond, Ivoclar Vivadent) was applied with a brush for 20 s, air-thinned for 3 s and photo-polymerized for 40 s using an LED polymerization unit (Bluephase, Ivoclar Vivadent) from a constant distance of 2 mm from the surface.

Dentin preparation

Buccal and lingual surfaces were trimmed (Isomet, Buehler Ltd., Lake Bluff, IL, USA) under water-cooling until flat dentin surfaces were achieved. Dentin level after flattening was considered as superficial dentin. One mm below this level was indicated and considered as deep dentin [16]. Dentin surfaces were then ground finished using 600-grit silicon carbide papers (Stuers A/S, Ballerup, Denmark) under water-cooling and then rinsed thoroughly in order to create bonding surfaces covered with smear layer [17]. Three-step etch-and-rinse adhesive system (Syntac, Ivoclar Vivadent) was used for dentin conditioning. First, primer (Syntac Primer, Ivoclar Vivadent) was applied using microbrushes for 30 s, air thinned gently with oil-free air. Then adhesive (Syntac Adhesive, Ivoclar Vivadent) was applied for 30 s, air thinned and finally bonding agent (Heliobond, Ivoclar Vivadent) was applied, air-thinned according to the manufacturer's instructions and photo-polymerized (Bluephase, Ivoclar Vivadent) for 40 s. Light intensity was assured to be higher than 1200 mW/cm², verified by a radiometer after every 8 specimen (Model 100, Kerr, Orange, CA, USA).

Bonding procedures for SBT, TBT, μ SBT

One calibrated operator carried out adhesive procedures throughout the experiments. Translucent polytetrafluoroethylene (Teflon) molds (DuPont, Saint-Gobain, France; for SBT: height: 4 mm, diameter: 2.9 mm; for TBT: height: 4 mm, diameter: 3 mm; for μ SBT: height: 4 mm, diameter: 0.8 mm) were stabilized on the enamel or dentin specimens in a custom made device. The mold was filled with the resin composite (Quadrant Universal AC, Cavex, Haarlem, The Netherlands, Shade A3), a metal pin was inserted to ensure 100 μ m thickness at the first layer of the increment and it was photo-polymerized (Bluephase, Ivoclar Vivadent). The mold was filled in two increments and polymerized for 40 s from 5 directions from a distance of 2 mm. Oxygen inhibiting gel (Oxyguard, Kuraray, Tokyo, Japan) was applied at the bonded margins and rinsed with copious water after 1 min.

Bonding procedures for μ TBT

Each tooth with exposed dentin surfaces was duplicated with resin composite (Quadrant Universal AC, Cavex) using a mold made out of condensation curing polysiloxane, putty soft consistency impression material (Alphasil Perfect, Müller-Omicon, Cologne, Germany). Resin composite was incrementally condensed into the mold and each layer was photo-polymerized (Bluephase, Ivoclar Vivadent) for 40 s. As a result, the bonding surface area of the resin composite blocks had the same surface area with the dentin surfaces. One composite resin block was fabricated for each tooth. Initially, the resin composite-dentin assembly was fixed with cyanoacrylate adhesive (Super Bonder Gel, Loctite Ltd., São Paulo, Brazil) on cylindrical metallic base of the cutting machine. The calibration of the machine was repeated for each new specimen. Bar specimens (sticks) were obtained by cutting the assembly using steel diamond discs (Accutom-50, Stuers A/S, Ballerup, Denmark) at low speed under water-cooling. The external sections of 1 mm were eliminated due to possible excess or absence of resin composite. The blocks were turned 90° and fixed again on the metallic base. Four transversal sections were obtained from each dentin-composite block and from those sections sticks with a length of ± 8 mm and adhesive area of ± 1 mm² were obtained. Thus, only the central specimens were used for the experiments. These sticks were examined under an optical microscope (Zeiss MC 80 DX, Jena, Germany) at $\times 50$ magnification and only those crack-free, structurally intact ones were selected for the experiments. In total, 52 sticks were obtained from enamel and 43 from dentin group. Bonding area of each stick specimen was measured before the tests using a digital caliper with an accuracy of 100 μ m.

Storage conditions

The specimens were stored in an incubator (Binder GmbH, Tuttlingen, Germany) at 37 °C for 24 h and then subjected to bond tests.

Macroshear and macrotensile tests

For the SBT, μ SBT, specimens were mounted in the jig of the Universal Testing Machine (Zwick ROELL Z2.5 MA 18-1-3/7, Ulm, Germany) and the shear force was applied using a shearing blade for SBT and a metal wire for μ SBT to the adhesive interface until failure

occurred. The load was applied to the adhesive interface, as close as possible to the surface of the substrate at a crosshead speed of 1 mm/min and the stress-strain curve was analyzed with the software program (TestXpert, Zwick ROELL, Ulm, Germany). For the TBT, specimens were mounted in the corresponding jig and resin composite disc was pulled with a grip from the substrate surface at a crosshead speed of 1 mm/min. For the μ TBT, the sticks were fixed to the alignment device with one drop of cyanoacrylate glue (Super Bonder Gel) on the resin composite and one on the dentin part of the bar specimen. It was made sure that the adhesive interface was free of the glue. The tensile force was applied at a cross-head speed of 1 mm/min until debonding.

Microscopic evaluation and failure type analysis

After adhesion tests, debonded specimen surfaces were analysed for failure types using an optical microscope (Zeiss MC 80 DX, Jena, Germany) at $\times 50$ magnification. Failure types were classified as follows: Score 1: Cohesive1: Cohesive failure in the substrate, Score 2: Mixed1: Combination of adhesive and cohesive failure types in the substrate and bonding agent, Score 3: Adhesive: Adhesive failure of bonding agent from the resin composite surface with no remnants on the resin composite, Score 4: Mixed2: Combination of adhesive and cohesive failure types in the bonding agent and resin composite, Score 5: Cohesive2: Cohesive failure in the resin composite.

Statistical analysis

According to the two-group Satterthwaite t-test (SPSS Software V.20, Chicago, IL, USA) with a 0.05 two-sided significance level, a sample size of 15 in each experimental group was calculated to provide more than 80% power to detect a difference of 7.45 MPa between mean values. Kolmogorov-Smirnov and Shapiro-Wilk tests were used to test normal distribution of the data. As the data were normally distributed, Univariate analysis of variance was applied to analyze possible differences between the groups where the bond strength was the dependent variable and substrate type (2 levels: enamel vs. dentin) and test methods (4 levels: SBT, TBT, μ SBT, μ TBT as independent variables). Interactions of substrate materials and test methods were analyzed using Tukey's or Dunnett-T3 *post hoc* tests. Following Anderson-Darling tests, maximum likelihood estimation without a correction factor was used for two-parameter Weibull distribution to interpret predictability and reliability of adhesion (Minitab Software V.16, State College, PA, USA) and a two-sided χ^2 was used to compare the results. Statistical analyses of failure types were made using χ^2 test. *P* values less than 0.05 were considered to be statistically significant in all tests.

Results

Pre-test failures during cutting procedures in μ TBT were considered as 0 MPa.

While test method ($p < 0.001$), substrate type ($p < 0.001$) significantly affected the bond results, interaction terms were not significant ($p = 0.237$).

When testing adhesion of resin composite to enamel, SBT (25.9 ± 5.7)^a, TBT (17.3 ± 5.1)^{a,c} and μ SBT (27.2 ± 6.6)^{a,d} test methods showed significantly higher mean bond values

compared to μ TBT (10.1 ± 4.4)^b ($p < 0.05$; Table 2). Adhesion of resin composite to dentin did not show significant difference depending on the test method (12 ± 5.7 – 20.4 ± 4.8 ; $p > 0.05$).

Only with SBT, significant difference was observed for bond values between enamel (25.9 ± 5.7) and dentin (12 ± 5.7 ; $p < 0.05$) while within each type of test method, mean bond strength to enamel and dentin did not show significant difference ($p > 0.05$; Table 3a–3c).

Weibull distribution presented the highest shape values for enamel-SBT (5.25)/ μ SBT (4.65) and dentin- μ SBT (4.86) among substrate-test combinations.

With μ TBT, pre-test failures were more commonly experienced with enamel than with dentin. Failure types showed significant differences between enamel and dentin ($p < 0.05$). Regardless of the test method, cohesive failures in substrate were more frequent in enamel (19.1%) than in dentin (9.8%).

Discussion

This study was undertaken in order to evaluate the adhesion of resin composite to enamel and dentin using macro- and micro-shear and tensile adhesion methods and to evaluate the failure types after debonding. Since both the substrate type and the test method significantly affected the bond strength results, the null hypotheses tested could be rejected.

In order to measure the bond strength values between an adherent and a substrate accurately, it is crucial that the bonding interface should be the most stressed region, regardless of the test methodology being employed. Previous studies using stress distribution analyses have reported that some of the bond strength tests do not appropriately stress the interfacial zone [18,19]. Shear tests have been criticized for the development of non-homogeneous stress distributions at the bonded interface, inducing either underestimation or misinterpretation of the results, as the failure often starts in one of the substrates and not solely at the adhesive zone [18,19]. Conventional tensile tests also present some limitations, such as the difficulty of specimen alignment and the tendency for heterogeneous stress distribution at the adhesive interface. On the other hand, when specimens are aligned correctly, the microtensile test shows more homogeneous distribution of stress, and thereby more sensitive comparison or evaluation of bond performances [20]. However, minute deviations in specimen alignment in the jig may cause increase bond strength due to shear component being introduced during debonding bonded joints [20]. According to the Griffith's theory [10], the tensile strength of the uniform materials decreases when the specimen size is increased. This outcome is a function of the distribution of defects in the material, since the larger bonded areas of the beams have more defects than smaller specimens. Overall, adhesion related studies in dentistry, bonded surface areas range from 3 mm² to 1 mm² in macro- and micro-test methods, respectively [9]. Due to the reduced bonded area and more homogeneous distribution of stresses, micro-test methods tend to show significantly higher bond strength results than the macro-test methods. This could eventually affect the ranking of materials being tested in one study [11]. To the best of our knowledge, no study exists to date where all four types of adhesion tests are employed in one study on both enamel and dentin.

Based on the results of this study, significantly higher results were obtained for bond strength of resin composite to enamel with SBT, TBT and μ SBT methods than with μ TBT. Interestingly, the smaller size of the bonded area did not necessarily resulted in higher

Table 2. The mean bond strength values (MPa \pm standard deviations) of SBT, TBT, μ SBT, μ TBT, Weibull modulus, distribution and frequency of failure types per experimental group analyzed after bond strength test: Score 1: Cohesive failure in the substrate, Score 2: Mixed1: Combination of adhesive and cohesive failure types in the substrate and bonding agent, Score 3: Adhesive: Adhesive failure of bonding agent from the resin composite surface with no remnants on the resin composite, Score 4: Mixed2: Combination of adhesive and cohesive failure types in the bonding agent and resin composite, Score 5: Cohesive2: Cohesive failure in the resin composite. The same superscript lowercase letters in the same column indicate no significant differences based on the substrate type and uppercase letters based on the test method ($p < 0.05$). For test group descriptions see Figure 1.

Group	Substrate	Test method	Produced/ Pre-test fail- ures/Final analyzed specimens	Bond strength (Mean \pm SD)	Min–Max (95% CI)	Weibull modulus m (95% CI)			Failure type distribution n (%)				
						m	Scale	CI	Score 1	Score 2	Score 3	Score 4	Score 5
1	Enamel	SBT	16/0/16	25.9 \pm 5.7 ^{aA}	11.5–33.6 (22.4–29.3)	5.25	28.1	(3.46–7.95)	3 (18.8)	0 (0)	11 (68.8)	2 (12.5)	0 (0)
2	Enamel	TBT	16/0/16	17.3 \pm 5.1 ^{a,cB,C}	10.1–27.1 (14–20.5)	3.78	19.1	(2.47–5.79)	4 (33.3)	0 (0)	2 (8.3)	7 (58.3)	0 (0)
3	Enamel	μ SBT	16/0/16	27.2 \pm 6.6 ^{a,dD}	17.6–37.3 (23.2–31)	4.65	29.7	(3.08–7.01)	0 (0)	0 (0)	6 (40)	6 (40)	3 (20)
4	Enamel	μ TBT	52/26/26	10.1 \pm 4.4 ^{b,E}	6–17.5 (4.9–15.2)	2.44	11.4	(1.32–4.51)	7 (17.1)	11 (26.8)	10 (24.4)	6 (14.6)	7 (17.1)
5	Dentin	SBT	16/0/16	12 \pm 5.7 ^{a,B,D,E}	3.4–22.12 (8.8–15.2)	2.22	13.6	(1.49–3.32)	0 (0)	0 (0)	16 (100)	0 (0)	0 (0)
6	Dentin	TBT	16/0/16	13.1 \pm 5.6 ^{a,B,E}	6.7–23.1 (9.7–16.5)	2.51	14.8	(1.67–3.77)	0 (0)	0 (0)	10 (71.4)	4 (28.6)	0 (0)
7	Dentin	μ SBT	16/0/16	20.4 \pm 4.8 ^{a,C,D,E}	9.4–26.5 (16.8–23.7)	4.86	22.2	(3.01–7.85)	0 (0)	0 (0)	9 (75)	2 (16.7)	1 (8.3)
8	Dentin	μ TBT	43/10/33	15.9 \pm 5.4 ^{a,B,E}	6.6–24 (9.3–22.3)	3.21	17.7	(1.66–6.23)	8 (25.8)	1 (3.2)	7 (22.6)	3 (9.7)	12 (38.7)

Table 3a. Significant differences between mean bond strengths of resin composite to enamel based on the test method (Tukey's and 2-sided Dunnett-T *post hoc* tests, $\alpha = 0.05$). For group descriptions see Figure 1.

Enamel	SBT	TBT	μ SBT	μ TBT
SBT	–	0.067	1.000	0.000
TBT	0.67	–	0.020	0.566
μ SBT	1.000	0.020	–	0.000
μ TBT	0.000	0.566	0.000	–

Table 3b. Significant differences between mean bond strengths of resin composite to dentin based on the test method (Tukey's and 2-sided Dunnett-T *post hoc* tests, $\alpha = 0.05$).

Dentin	SBT	TBT	μ SBT	μ TBT
SBT	–	1.000	0.085	0.970
TBT	1.000	–	0.245	0.996
μ SBT	0.085	0.245	–	0.946
μ TBT	0.970	0.996	0.946	–

Table 3c. Cross-comparison of significant differences between mean bond strengths of resin composite for enamel vs. dentin based on the test method (Tukey's and 2-sided Dunnett-T *post hoc* tests, $\alpha = 0.05$).

Enamel vs. Dentin	SBT	TBT	μ SBT	μ TBT
SBT	0.000	0.000	0.564	0.114
TBT	0.624	0.865	0.981	1.000
μ SBT	0.000	0.000	0.297	0.046
μ TBT	1.000	0.993	0.129	0.901

bond strength, namely both SBS (25.9 MPa) and μ SBT (27.2 MPa) conveyed similar results, also supported by Weibull moduli with 5.25 and 4.65, respectively. Although μ TBT offers bonded areas of 1 to 1.2 mm², the complex nature of specimen preparations yields to pre-test failures [21]. In this study, the lost specimens during cutting procedures, were considered as 0 MPa to represent the worse-case scenario during statistical analysis. In some studies, such debonded specimens were completely excluded from statistical analysis yielding to higher bond strength results. In fact, pre-test failures could be indicative for less favourable bond strength. However, this statement has to be connected to the substrate type in that bond strength results were favourable with all three test methods (SBT, TBT and μ SBT) but not μ TBT with the same adhesive and resin composite combination. Moreover, the incidence of pre-test failures with enamel was more common than with dentin. This could be also attributed to the high hardness of enamel (270–350 KHN) compared to dentin (50–70 KHN) [22] which caused deflexion of the substrate from the composite block during cutting procedures, which was not related to the bond strength. It also has to be noted that in this study, neither the composite block nor the whole tooth was secured in acrylic [21]. Thus, this approach could be considered as a worse case scenario, when testing adhesion of resin materials to dentin.

In general in adhesive dentistry, adhesion values to enamel are considered as gold standard as the etched enamel surface provides excellent micromechanical retention. Yet, it has to be realized that enamel is a crystalline substance that consists of hydroxyapatite arranged in prisms that comprises 96 wt% inorganic matter, 0.4–0.8 wt% organic matter such as proteins, lipids, carbohydrates or lactate and 3.2–3.6 wt% water [1] and the histological

structure of these hydroxyapatite crystals of enamel in cross section is hexagonal. From lateral perspective, they appear as small rods, of which each is built out of about 100 crystals [2]. However, they may also appear as prisms and in the centre of the prisms, the crystals are placed parallel to the longitudinal axis and in the outer parts in almost 90° inclination [2]. This change in direction gives the prisms a honeycomb shape structure and the interprismatic areas consist of more loosely packed and randomly oriented crystals surrounded by a higher quantity of water and inorganic matter. Thus, enamel microstructure is in fact not a homogeneous structure and anatomical variations could be observed on enamel surface also sometimes due to the presence of aprismatic enamel layer [2].

Using conventional etch-and-rinse adhesive approach selectively dissolves hydroxyapatite crystals through etching with 37% H_3PO_4 followed by polymerization of resin that is readily absorbed by capillary reaction within the created etch prisms [23]. Adhesive system used in this study was never tested in conjunction with TBT and μ SBT on enamel. However, our results with SBT, comply well with the findings of other studies (21.6 ± 5.8 – 29.2 ± 7.3 MPa) in combination with other resin composites [24–28] except with one study where higher mean value was reported (42.9 ± 9 MPa) [23]. μ TBT results for enamel could be compared with only one study where higher mean bond strength was reported (38.9 ± 9.2 MPa) [29]. In that study, pre-test failures were not involved in statistical analysis and similar to this study, more frequent microcracks were observed in enamel than in dentin that was also attributed to flaw introduction during preparation [30].

Similar to adhesion to enamel, bonding to dentin was achieved using an etch-and-rinse adhesive approach where hydroxyapatite crystals are selectively dissolved that is followed by resin polymerization. Unlike enamel, dentin consists only of about 68% inorganic hydroxyapatite where the rest is mostly organic collagen fibers. The primary bonding mechanism to dentin is primarily diffusion based and depends highly on hybridization or infiltration of resin within the exposed collagen fiber scaffold. Thus, true chemical bonding to dentin is fairly unlikely since the functional groups of monomers have only weak affinity to the hydroxyapatite-depleted collagen [23]. As a result of the higher organic fraction and other specifications dentin bonding is much more complex and therefore more technique sensitive than enamel bonding. Over etching or over drying dentin could also lead to collapse of collagen fibers and thereby weaken bond strength [29]. In this study, selective etching approach was employed for dentin using mild maleic acid (Syntac Primer) and subsequently dentin was rehydrated with adhesive resin (Syntac Adhesive) that is water-based. In the dentin group, the test method did not significantly affect the results. However, μ SBT showed more reliable Weibull modulus with 4.86 compared to those of other test methods (2.22–3.21). No μ SBT results could be found in the literature with the adhesive system tested. However, with SBT (10.2 – 19.45 ± 5.04) [28,31–34] and with TBT wide ranges of mean values were reported (3.89 ± 3.47 – 23.8) [28,31–34]. One possible explanation for this wide range could be attributed to the resin composite used as the elasticity modulus of the materials show variations in different studies. Nevertheless, with the exception of μ SBT (4.86), overall Weibull moduli for adhesion to dentin (2.22–3.21) was lower than for enamel. Similar moduli were reported for SBT and TBT using the same adhesive system [35,36].

In this study, adhesion procedures were performed complying with ISO/TS 11405 specifications [15] that are frequently disregarded in adhesion studies. In this regard, one important aspect in bonding to dentin is the density and orientation of dentin tubuli. Accordingly, buccal dentin was used as a substrate according to the specifications. However, when occlusal

dentin is used as a substrate and perfusion simulations are performed, significantly lower results could be obtained to dentin especially in deep dentin closer to the pulp with SBT (8 ± 3.7) or TBT ($2.6 \pm 1.4 - 5.08 \pm 3.69$) tests [24,26–42].

A review study of Scherrer et al. in 2010 revised studies looking at dentin bond strength and not enamel. The results of this study, indicated that microtensile test results in less mean values on enamel than on dentin due to microfractures that occurred during stick cutting procedures in the former [43]. In this study, all pre-test failures were considered as 0 MPa. Conversely, since the bonded area is larger in macrotensile tests, lower values could have been expected. Yet, the lack of pretest failures due to the nature of the macrotensile test, namely bonded specimens are only subjected to tensile forces without any cutting procedures, macrotensile test delivered higher results. However, in the review of Scherrer et al, the reviewed material did not include the pretest failures as zero value but excluded them from statistical analysis [43]. This might have caused the discrepancy in the mean values between the reported values and those of ours. It has to be noted however that in the review of Scherrer big variation was noted between the results of the reviewed papers [43]. On the contrary, our results, even though pretest failures were recorded as zero values, presented lower standard deviation that could be also traced in the Weibull statistics.

Bond strength results in adhesion studies should be also interpreted with failure types. Cohesive failures in the substrate (Score 1) and combination of adhesive and cohesive failure types in the substrate and bonding agent (Score 2) indicate that bond strength of the adhesive system and the resin composite exceeds that of the cohesive strength of the substrate. Regardless of the test method, the incidence of Score 1 and Score 2 were more frequent in enamel than in dentin. Thus, when these two failure types are considered, adhesion to enamel could be considered more reliable than to dentin. Although the focus was on the adhesion of the resin based materials to enamel and dentin, it has to be noted that bond strength of the adhesive resin to the resin composite also plays a significant role in interpreting failure types. Score 3,4 and 5 are also influenced by the adhesive-composite adhesion. The incidence of Score 5 that is the cohesive failure in the resin composite was almost only experienced with μ TBT method for both enamel and dentin. Thus, this type of score reveals that adhesion to both enamel and dentin exceeded that to the resin composite. In that respect, μ TBT indicates that adhesion is reliable to the both substrates at least with the tested specimens left after pre-test failures.

Clinical conditions during chewing functions expose restorative materials to multiple strains in different directions that could be a combination of both shear and tensile. Fracture toughness test and interpretation of fracture mechanics was recently considered as an alternative to other bond measurement methods as it considers the visco-elastic nature of the tested materials better than the commonly used bond strength methods. Unfortunately, the preparation technique is usually more complex than most bond tests and also the stresses presented within the adhesive resin are quite complex [44]. The overabundant number of adhesive resin and resin-based composites in restorative dentistry would possibly continue to be tested and ranked prior to clinical trials. Due to technique sensitivity in specimen preparation, only one test method could not be advised for adhesion studies in dentistry. Hence, ranking of materials could be made based on the research question where μ SBT could be considered less technique sensitive and μ TBT could be used for testing worse case scenarios. Future studies should also involve pulp pressure, the use of disinfectants and the effects of possible contaminants such as provisional cements especially on dentin [45,46].

Conclusions

From this study, the following could be concluded:

- (1) Adhesion of resin composite to enamel was significantly higher with SBT, TBT and μ SBT methods than with μ TBT but adhesion to dentin did not show significant difference depending on the test method.
- (2) Only with SBT, significant difference was observed for bond values between enamel and dentin.
- (3) Weibull distribution showed more reliable adhesion of the resin composite to enamel-SBT/ μ SBT and dentin- μ SBT compared to substrate-test combinations.
- (4) μ TBT resulted in frequent pre-test failures more commonly with enamel than with dentin. Regardless of the test method, cohesive substrates in substrate were more frequent in enamel than in dentin, indicating more reliable adhesion to enamel.

Clinical relevance

Based on the bond strength values, Weibull modulus and the failure types, adhesion to enamel is more reliable than to dentin. μ SBT test could be considered more suitable for testing adhesion of resin-based materials to enamel or dentin.

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